AMENDMENT NO. 1 DECEMBER 2022

TO

IS 17440 : 2020 CARBON BLACK — SPECIFICATION

(*Foreword*) — Add a new para after para 3:

‘For alternate test methods, assistance has been derived from various ASTM Standards such as ASTM D1765, ASTM D1512, ASTM D3265, ASTM D1510, ASTM D3324, ASTM D6556, ASTM D1508, ASTM D1509, ASTM D1506, ASTM D1513, ASTM D1514, ASTM D1618 and ASTM D2414.’

(*Foreword*) — Substitute para 4 with the following:

‘The composition of Committee responsible for the formulation of this standard is given at Annex Q.’

(*Page* 1, *clause* **1**) — Substitute the existing with the following:

‘1 SCOPE

This standard prescribes requirements and methods of test for pelleted furnace grade carbon black for varied applications such as, but not limited to paints, plastics, inks, tyres and rubber products. Any type of regenerated/recovered carbon black is not covered within this scope.’

(*Page* 1, *clause* **4.1**) — Substitute the existing with the following:

‘4.1 Packaging

The material may be supplied in bags or any other packing as agreed to between the manufacturer and supplier. In case of bag packing, the net mass of each bag shall be 5 kg to 25 kg with a tolerance of ± 2 percent. The bags shall be shaped to facilitate stacking in pellets. Material can also be in FIBC (Flexible Intermediate Bulk Container) bags of net capacity varying from 500 kg to 1 300 kg as per mutual requirement subject to compliance to statutory regulations and legal metrology rules.’

(*Page* 2, *Table* 1) — Substitute the existing Table 1 with the following:

‘Table 1 Requirements for Carbon Black
(*Clause* 3)

Sl No.	Characteristic	Grade I	Grade II	Grade III	Grade IV	Grade V	Grade VI	Grade VII	Annex
i)	Ash Content (percent by mass), *Max*	1.0	1.0	1.0	1.0	1.0	1.0	1.5	A
ii)	*p*H Value	4-10	4-10	4-10	4-10	4-10	4-10	1-10	B
iii)	Tinting Strength (percent)	105-150	105-135	70-135	Not applicable	Not applicable	Not applicable	> 35	D
iv)	Pour Density (kg/m^3)	270 -410	280 -490	280 -500	280 - 410	320 - 520	290 - 550	Not applicable	E

Price Group 6

v)	Iodine Adsorption No. (mg/g or g/kg)	95 - 170	95 - 160	50 - 120	35 - 105	25 - 45	15 - 38	>15	F
vi)	Nitrogen Surface area (m^2/kg)	100-160	95 - 130	55 - 105	30-85	25 - 45	15 - 38	>15	H
vii)	Sieve residue on 45 micron, percent by mass, *Max*	0.100	0.100	0.100	0.100	0.100	0.100	0.100	J
viii)	Sieve residue on 500 micron,percent by mass, *Max*	0.001	0.001	0.001	0.001	0.001	0.001	0.001	J
ix)	Loss on Heating, percent by mass, *Max*	3.00	3.00	2.50	2.50	2.50	2.50	15.00	K
x)	Toluene Discolouration, percent Transmittance, *Min*	70.00	70.00	70.00	70.00	70.00	50.00	50.00	M
xi)	Fines Content,percent by mass, *Max*	12.00	12.00	12.00	12.00	12.00	12.00	15.00	N

Table 2 Optional Requirements for Carbon Black

(*Clause* 3)

12	Acetone Extract (percent by mass), *Max*	As agreed between the interested parties	C
13	Particle size determination (millimicrons), *Max*	As agreed between the interested parties	G
14	Oil absorption number (OAN), ml/100 g	As agreed between the interested parties	P

(*Page* 4, *Annex* B) – Add a new clause after **B-5**:

‘B-6 ALTERNATE METHOD

B-6.1 Outline

This test method covers the determination of *p*H value of carbon black.

B-6.2 Apparatus

B-6.2.1 *pH Meter* ─ having an accuracy of 0.05 *p*H and equipped with a combination electrode and RNC connector.

B-6.2.2 *Hot Plate*

B-6.2.3 *Spatula*

B-6.2.4 *Beaker* (100 ml)

B-6.2.5 *Watch Glass*

B-6.2.6 *Analytical Balance* — 0.01 g accuracy.

B-6.2.7 *SS Mug with Lid*

B-6.2.8 *Mill, mixer or mortar and pestle*

B-6.2.9 *Magnetic stir plate and bar*

B-6.3 Reagents

B-6.3.1 *DM Water/Distilled Water*

B-6.3.2 *Buffer Solution*, of 4.00, 7.00 and 9.18 *p*H value.

B-6.3.3 *Acetone*

B-6.4 Procedure (Method A – Boiling Slurry)

Pulverize pelleted or lumpy carbon black to a fine powder by using either high speed mixer or mortar and pestle. Boil the distilled water in a SS mug on hot plate without cover. Weigh 5 g of carbon black to the nearest 0.1 g into a 100 ml Beaker/10 gm sample in 100 ml water. Add 50 ml of boiled distilled water in the beaker having carbon black sample and add 2-3 drops of acetone to facilitate wetting of sample. Cover the beaker with watch glass and boil the mixture for 15 min, but do not allow all the liquid to evaporate. Cool the mixture to room temperature in an atmosphere free from chemical fumes which might contaminate the sample. Switch on the *p*H meter and standardize the *p*H Meter with buffer solutions, rinse the electrode with distilled water and wipe clean after each test. Place the beaker containing carbon black mixture in contact with *p*H meter electrode. Rotate it gently using the stir bar, place the electrode carefully so that the stir bar does not touch the electrode. When constant reading of *p*H is obtained, note down the *p*H from the screen, to the nearest 0.05. Rinse the electrode with distilled water and wipe clean. Keep the electrode soaking in distilled water when not in use.

B-6.5 Calculation

B-6.5.1 *pH Meter Final Reading.*

B-7 In case of dispute, test method as prescribed at **B-6** shall be the referee method.'

(*Page* 7, Annex D) — Add a new clause after **D-4.5**.

D-5 ALTERNATE METHOD

D-5.1 Outline

This test method covers the determination of tint strength of carbon black.

D-5.2 Apparatus

D-5.2.1 *Automatic Muller*

D-5.2.2 *Tint Tester*

D-5.2.3 *Spatulas*

D-5.2.4 *Analytical Balance*

D-5.2.5 *Oven*

D-5.3 Reagents

D-5.3.1 *Plasticizer* — for example Green Flex/Paraplex G-62

D-5.3.2 *Zinc Oxide (Lab/Analytical Grade)*

D-5.3.*3* *Acetone*

D-5.3.4 *ITRB (Industry Tint Reference Black)*

D-5.4 Procedure

Dry the carbon black sample, zinc oxide and ITRB sample at 125 ± 1 °C for 1 h and cool in a desiccator at room temperature. Weigh exactly 0.100 0 g of carbon black sample on a weighing paper and 3.750 0 g of zinc oxide. For, N-500, N-600 and N-700 series carbon blacks weigh exactly 0.200 0 g of carbon black and 3.750 0 g of zinc oxide. Using the syringe, place 2.20 ± 0.02 ml or weigh 2.20 g of plasticizer on a weighing paper and transfer it to the center of the mulling plate.

NOTE ─ If the use of 2.20 ml (2.20 g) of plasticizer produces an unmanageable fluid paste, then it is acceptable to use 2.00 ml (2.00 g) of plasticizer for both the ITRB standard and the sample.

Place the zinc oxide and carbon black in the center of the pool of plasticizer. Mix the three materials with a spatula, confining them as close to the center as possible; mix until they are well incorporated. Clean the material from the spatula onto the top of the mulling plate, maintaining a minimum loss of material.

Set the Muller for 25 revolutions with one extra mass on the arm equivalent to the force of 445 N (100 lbf) on the plate. Close the plates raise the mass arm and start the Muller. At the end of the 25 revolutions cycle, lower the mass arm and open the plates. Scrape the paste from the upper plate with a spatula to remove as much paste as possible and transfer it to the center of the lower plate. Then, with the lower plate rotating use the spatula to spread the paste to a flattened circle on the plate. Then work all of the paste to exact the center. Clean the material from the spatula onto the top mulling plate. Weigh exactly 0.100 0 g of ITRB on a weighing paper and 3.750 0 g of zinc oxide. Make a paste with plasticizer same as carbon black sample following above instruction. Repeat these steps three times more for a total of four cycles of 25 revolutions each. Remove the paste to a smooth, clean surface and clean the Muller plates using a solvent and wiping tissue paper. Clean the glass plate with a wiping tissue to remove any dust particles or film. Place a portion of the paste on the top edge of a glass plate with a spatula. Draw down the paste using a film applicator applying moderate consistent pressure. Draw down the paste to the bottom edge in 2 s to 3 s.

Pick up the film applicator without removing the excess paste adhered to it. Bring it to the beginning of the drawdown, and again draw down the paste to the bottom edge of the glass plate in 2 s to 3 s. Drawdown shall have a uniform surface approximately 65 mm wide (2.5 inches).

NOTE ─ If the surface is not uniform, prepare another drawdown.

Turn on the power switch of the tint tester and allow for 30 min warm-up. Place the reflectance head on the black calibration panel and adjust the digital readout to 0.00 using the ‘zero’ control potentiometer. Disregarding the first 75 mm (3 inch) at the top of the drawdown, set the reflectance head aperture over the drawdown of the ITRB 100 percent paste. Adjust the reflectometer to read an average of 3.00 for at least four readings taken at different positions. The instrument is now correctly adjusted for reading other pastes. The value of 3.00 is used for ITRB for making calculations. Determine the sample paste reflectance value.

Remove the paste and clean the drawdown area when all readings have been made. Determine the reflectance value of the prepared paste of the test sample by drawing down the film of each paste and with the help of a reflectometer.

D-5.5 Calculation

Tint Strength, percent = (I/S) × 100

where

I = Reflectance of existing Industrial Tint Reference Black
S = Reflectance for sample

(For the N-500, N-600 & N-700 series of Carbon black, divide the above results by 2)

NOTE — Respective SRB's for different grades of carbon black (or other available certified reference material) may be used for verification of the test results.

D-6 In case of dispute, test method as prescribed at **D-5** shall be the referee method.'

(*Page* 8, *Annex* F) — Add a new clause after **F-5**:

F-6 ALTERNATE METHOD

F-6.1 Outline

This test method covers the determination of iodine adsorption number of carbon black.

F-6.2 Apparatus

F-6.2.1 *Automatic Burette*

F-6.2.2 *Conical Flask*

F-6.2.3 *Beakers*

F-6.2.4 *Centrifuge Machine*

F-6.2.5 *Pipette* (20 and 25 ml)

F-6.2.6 *Oven*

F-6.2.7 *Desiccator*

F-6.3 Reagents

F-6.3.1 *Sodium Thiosulphate*

F-6.3.2 *Iodine*

F-6.3.3 *Potassium Iodide*

F-6.3.4 *Sodium Bicarbonate*

F-6.3.5 0.*1 N Potassium Dichromate Solution*

F-6.3.6 *Starch*

F-6.3.7 25 *percent HCl Solution*

F-6.4 Preparation of Sodium Thiosulphate Solution (0.039 4 N)

Weigh 19.562 5 g of sodium thiosulphate to the nearest of 0.1 mg. Transfer it to 2 litre volumetric flask. Add approximately 500 ml of DM water and 10 cm^3 or (10 ml) of n-amyl alcohol to the flask, shake well to dissolve sodium thiosulphate. Make up the volume up to 2 l mark by adding DM water. Place the flask on the magnetic stirrer and stir for 1 to 2 h. Age for 1 to 2 days, then stir for 0.5 h before using.

F-6.5 Standardization of Sodium Thiosulphate Solution

Weigh 3 g of potassium iodide and 2 g of sodium bicarbonate and transfer it to 250 ml conical flask. Add 20 ml DM water, 20 ml (0.1 N) potassium dichromate solution and 20 ml of 25 percent HCl. Titrate sodium thiosulphate solution against the above solution. When the content of conical flask becomes pale yellow, add 2-3 drops of starch solution. Addition of starch will make this solution blue-violet in color. At the end point the addition of last one drop of sodium thiosulphate solution will make the conical flask solution sea green in color.

Strength of sodium thiosulphate solution = $\frac{0.1 \times 10}{\text{Burette reading}}$

If N is not equal to 0.0 394 ± 0.000 1 adjust the solution in the following manner: if the solution is too strong, add water (2.5 cm^3/dm^3 solution for each 0.000 1 N over 0.0 394); if the solution is too weak, add solid sodium thiosulfate (0.025 g/dm^3 for each 0.000 1 N under 0.039 4).

In the absence of any ageing data of this standard solution, user to take care based on the usage. Also it is recommended to run standard iodine sample on a daily basis before use of the solution.

F-6.6 Iodine Solution (0.473 N)

Weigh 60 g iodine and 570 g potassium iodide and transfer them to a 1 000 ml Beaker. Add appropriate DM water, stir with glass rod to dissolve both the chemicals. Pour this solution in to 1 000 ml volumetric flask. Make up the volume to 1 000 ml mark by adding more DM water.

Prepare the desired amount of working iodine solution (0.047 3 N); use volumetric pipettes and volumetric flasks to prepare exactly 1 : 10 dilutions.

Standardization of Iodine Solution:
Take 20 ml of iodine solution and titrate against standard sodium thiosulphate solution.

$$\text{Normality of iodine solution N} = \frac{(0.0\ 394\ ) \times \text{titrate value}}{20}$$

If N is not equal to 0.047 3 N, adjust solution in the following manner: if the solution is too concentrated, add water (2.1 cm^3 or 2.1 ml) per dm^3 iodine solution for each 0.000 1 N over 0.047 3); if the solution is too diluted, add iodine (12.7 mg iodine per dm^3 or 12.7 ppm iodine solution for each 0.000 1 N under 0.047 3). Put a tag on solution bottle with strength and date of preparation.

F-6.7 Potassium Dichromate Solution (0.1N)

Weigh 4.903 5 g potassium dichromate previously dried at 125 °C ± 3 °C and cool. Transfer it to 1 000 ml volumetric flask. Dissolved it in DM water and make up the volume to 1 000 ml mark by adding more DM water.

F-6.8 Starch Indicator Solution

Add approximately 1 g of starch powder into 250 ml conical flask having approximately 100 ml hot DM water. Boil it for few minutes on hot plate and allow to cool.

F-6.9 Procedure

Dry the sample of carbon black in an oven at 125 ± 3 °C for 1 h. Cool it in a desiccator at room temperature. Weigh a mass of the dried sample into a glass vial as shown by the following table:

Iodine Number	*Sample Mass (g)*	*Ratio I_2 : Sample Mass*
0–130.9	0.500	50 : 1
131.0–280.9	0.250	100 : 1
281.0–520.9	0.125	200 : 1
521.0 and above	0.062 5	400 : 1

Transfer it into vials. Add 25 ml of standardized working 0.047 3 N iodine solution into the vials containing appropriate amount of sample and close it with rubber cork. Shake it approximately for 1 min and then centrifuge it for nearly 1 min. Decant it into 100 ml Beaker. Pipette 20 ml of this decanted solution in a 250 ml conical flask. Titrate it against sodium thiosulphate solution till pale yellow color appears. Add 2 to 3 drops of starch indicator solution to the content of conical flask when it becomes pale yellow in color. Addition of starch solution will develop blue-violet color. Again titrate it with sodium thiosulphate solution. At the end point, blue-violet color will disappear with the addition of last one drop of sodium thiosulphate solution.

NOTE — The burette reading to determine the volume of sodium thiosulphate solution required for sample.

Make a blank iodine titration taking 20 ml of 0.0473 N iodine solution and note down the reading. Find out the difference of blank value and sample titration value and then note the final reading.

F-6.10 Calculation

Calculate the iodine adsorption number to the nearest 0.1 mg/g (g/kg) as follows:

$$I = \frac{B-S}{B} \times \frac{V}{W} \times N \times 126.91$$

where

I = Iodine adsorption number, mg/gm of carbon black
B = ml of sodium thiosulphate solution required for the blank
S = ml of sodium thiosulphate solution required for the sample
V = Calibrated volume of 25 ml iodine
W = gram of carbon black sample
N = normality of iodine solution
126.91 = equivalent mass of iodine

NOTE ─ For laboratories using automatic/electronic/software enabled pipettes/dispensers and burettes, the above solution and principle of testing will apply. Users will have to follow the standard instruction manual of the respective equipment they may be using.

Respective INR (or other available certified reference material) samples are also tested for iodine numbers to establish the validity of the test method. The obtained values are used to create normalization curve and normalize the values obtained on the analyzed samples.

F-7 In case of dispute, test method as prescribed at **F-6** shall be the referee method.'

(*Page* 12, *Annex* H) — Add a new clause after **H-6.3**:

'H-7 ALTERNATE METHOD

H-7.1 Outline

This test method covers the determination of NSA of carbon black sample by BET Multipoint.

H-7.2 Apparatus

H-7.2.1 *Surface Area Analyzer*

H-7.2.2 *Sample Cell with Filler Rods*

H-7.2.3 *Dewar Flask*

H-7.2.4 *Heating Mantle*

H-7.2.5 *Vacuum Pump*

H-7.2.6 *Oven*

H-7.2.7 *Analytical Balance*

H-7.2.8 *High Vacuumgrease*

H-7.2.9 *Petri Dish*

H-7.2.10 *Desiccator*

H-7.3 Reagents

H-7.3.1 *Liquid Nitrogen*

H-7.3.2 *Nitrogen Gas*

H-7.4 Procedure

Take appropriate quantity of sample, dry it in oven at 125 °C for 1 h. Take 1 g of sample in bulb type sample cell and insert filler rod in the sample cell. Maintain the temperature 300 °C of analysis station(s). Insert the sample cell inside the heater and close it with clamp then fix the cells in both of the stations station A and B. Start the vacuum degas for 1 h at 300 °C. After degassing unload sample cells maintain the temperature to the room temperature and cool the sample cells to the room temperature. Weigh 0.400 0 g of sample from the degassed sample and start the analysis. Fix the sample cells in the analysis station(s). Fill the dewar flask with the liquid nitrogen and place it below the sample cells and start the analysis. When the analysis completed note NSA and STSA value directly from the machine.

H-7.5 Calculation

Record the result as recommended by the equipment software.

H-8 In case of dispute, test method as prescribed at **H-7** shall be the referee method.'

(*Page* 13, *Annex* K) — Add a new clause after **K-5**:

'**K-6 ALTERNATE METHOD**

K-6.1 Outline

This test method covers the determination of heating loss of carbon black sample.

K-6.2 Apparatus

K-6.2.1 *Oven*

K-6.2.2 *Glass Petri Dish with Cover/Weighing Bottle with the Stopper*

K-6.2.3 *Analytical Balance* (0.1 mg)

K-6.3 Procedure

K-6.3.1 *Heat Loss by Oven Drying Method*

Dry the petri dish/weighing bottle and the stopper with cover at 105 ± 3 ^{0}C / 125 ± 3 ^{0}C for 30 min in an oven. Cool the petri dish/weighing bottle with cover in desiccators. Weigh it to the nearest 0.1 mg. Weigh 2 g of carbon black sample into the petri-dish/weighing bottle to the nearest 0.1 mg and put it in the air oven maintained at 125 ± 3 ^{0}C for 1 h with cover/stopper removed. Remove the petri dish/weighing bottle with cover from oven and place in the desiccators for cooling. Weigh the petri dish/weighing bottle with sample after cooling.

K-6.3.2 *By Moisture Analyzer*

Start the moisture analyzer. Set the temperature at 125 ^{0}C. Create the identification of sample in moisture analyzer. Put approximately 2 g of sample in the pan of the moisture analyzer. Press the start button. Note down the reading when beeps start from moisture analyzer.

F-6.4 Calculation

Heat loss, percent by mass = $\frac{(B-C)}{B-A} \times 100$

where

A = Weight in g of petri dish and cover
B = Weight in g of petri- dish, cover and sample before heating
C = Weight in g of petri- dish, cover and sample after heating

K-7 In case of dispute, test method as prescribed at **K-6** shall be the referee method.’

(*Page* 15, *Annex* N) — Add a new clause after **N-4**:

‘**N-5 ALTERNATE METHOD**

N-5.1 Outline

This test method covers the determination of fines and attrition of carbon black

N-5.2 Apparatus

N-5.2.1 *Sieve Shaker*

N-5.2.2 125 *Mesh Sieve*

N-5.2.3 *Receiving Pan*

N-5.2.4 *Analytical Balance* — 0.000 1g accuracy.

N-5.3 Procedure

N-5.3.1 Stack the sieves from bottom to top as, bottom receiving pan and 125 mesh sieve. Weigh 25 g carbon black sample to the nearest of 0.1 g and transfer it on 125 mesh sieve. Put the sieve assembly on the sieve shaker and shake it for 5 min. Weigh the material passed through 125 mesh and retained on the receiving pan.

N-5.3.2 *For Attrition*

After removing fines powder from receiving pan again put the assembly on the sieve shaker and shake for another 15 min. Stop the machine. Remove fines powder from receiving pan and weigh.

N-5.4 Calculation

Fines, percent by mass = Weight of material on receiving pan (in step N-5.3.1) × 4
Attrition, percent by mass = Weight of material on receiving pan (in step N-5.3.2) × 4

N-6 In case of dispute, test method as prescribed at **N-5** shall be the referee method.’

(*Page* 15, *Annex* P) — Add a new annex after Annex N:

‘ANNEX P

(*Clause* 3, *Table* 1)

TEST METHOD OF OIL ABSORPTION NUMBER (OAN)

P-1 OUTLINE

This test method covers the determination of oil absorption number (OAN) of carbon black.

P-2 APPARATUS

P-2.1 Absorptometer — equipped with constant rate burette that delivers 4 ± 0.024 ml/min.

P-2.2 Analytical Balance — 0.01 g accuracy.

P-2.3 Spatula

P-2.4 Petridish

P-2.5 Oven

P-2.6 Desiccator

P-2.7 Stainless Steel Beaker — 150 ml.

P-3 REAGENTS

P-3.1 DBP oil/Epoxidised fatty acid ester (EFA) oil/Paraffin oil

P-3.2 Standard Reference Black

P-4 PROCEDURE

Dry carbon black sample at 125 ± 3 °C for 1 h and cool in a desiccator at room temperature. Follow instructions of user manual based on make and type of the burette. Ensure burette filling and charging system is free of air bubbles. Weigh 20 g (25 g for low structure black with typical OAN values less than 80 or otherwise recommended for standard blacks) of carbon black to the nearest of 0.01 g. Add the weighed carbon black sample into the tightly fixed mixing chamber and start the machine. Suitable steps for starting the machine depend on the make of the aborptometer and its software if any. Follow the operating instruction of the aborptometer suitably. Necessary precaution to be taken for tread black and carcass black based on the make of the equipment. The absorptometer and dozing stop when maximum torque is attained. Note down the reading of the OAN value as per operations instructions of the equipment manual. The final OAN value detection will depend on the make and type of equipment. Open and clean the chamber to get ready for the next test.

P-5 RECORD

Record the result as per the practice of the laboratory.

P-6 EQUIPMENT READINESS

Follow the practice of mixing chamber readiness, normalization, and calibration technique based on the type and make of absorptometer. Use standard reference material as per recommendation. Refer to equipment operating manual as required.

P-7 CALCULATION

P-7.1 Method A (Manually Operated Absorptometers)

Note down the reading of the burette (in ml) at which the dozing stops on attainment of maximum torque (end-point).

OAN Value, ml/100 gm = $\frac{\mathrm{A}}{\mathrm{B}} \times 100$

where

A = burette reading in ml, second decimal place
B = weight of the sample in g, second decimal place.

P-7.2 Method B (Software Operated Absorptometers)

For software operated equipment, no manual calculation is required. The OAN value is as reported by the software, based on the nature of the sample (hard grade or soft grade) and the make of the equipment. Note down the software enabled reading and report accordingly. The principle of OAN being reported is as:

OAN Value, ml/100gm = $\left(\frac{A}{B} \times 100 \times C\right) + D$

where

A = burette reading in ml, second decimal place
B = weight of the sample in gm, second decimal place
C = slope of normalization curve
D = y-intercept of normalization curve.

Normalization technique should be followed using standard reference black as mentioned in **P-6**.’

(*Page* 16) — Substitute ‘ANNEX Q’ for ‘ANNEX O’.

(CHD 1)

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